APPLICATION NOTE

Rigaku oxford diffraction

Improving data quality for a highly absorbing mineral, Hereroite, with the PhotonJet™ Ag source

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Introduction

When collecting X-ray data on samples containing several heavy elements that are densely packed, for example; minerals, crystalline alloys, metal clusters and other inorganic materials such as perovskites, absorption can be an issue. The total absorption (μ) of any sample is dependent on the sample composition, packed sample density, the size of the crystal, and the X-ray wavelength. By reducing the latter you can lower the overall absorption and get improved data quality. For home-lab instruments considerable benefits can be found for these types of samples by employing silver radiation which has a shorter wavelength (0.56 Å).

In order to best utilize the shorter wavelength, a CdTe HPC detector was used. CdTe is better suited to absorb high energy X-rays and thus for Ag Kα experiments provides excellent performance.

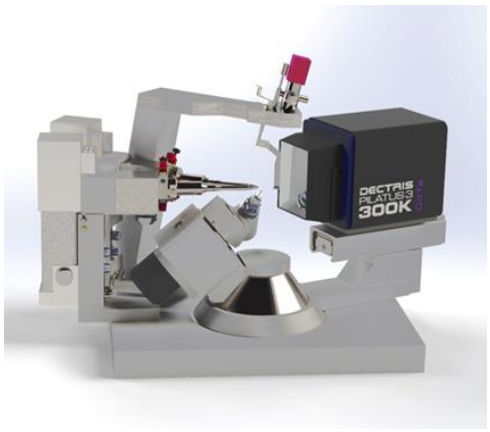

Figure 2. XtaLAB Synergy-S with the PILATUS3 R CdTe 300K detector

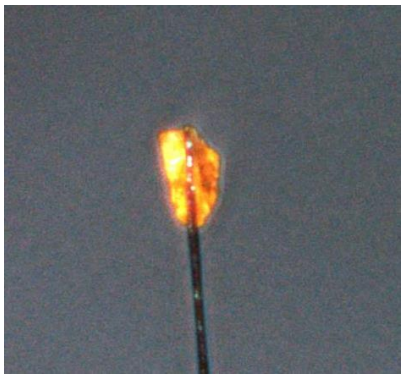

Figure 1. Crystal sample
μ (Ag) value of 47.5 mm^{-1}
(0.10 x 0.08 x 0.03 mm)

The mineral, Hereroite, has a μ value close to 100 mm^{-1} when using Mo Kα radiation. With silver radiation the μ value is approximately halved which yields substantially improved data quality.

The sample in Fig. 1 was previously measured on an older SuperNova instrument with the first generation Silva source and a CCD detector. In this application note this same crystal was remeasured in order to quantify the improvements which can be made with the current generation of instrument, the XtaLAB Synergy-S, equipped with the PhotonJet™ Ag source and the PILATUS3 R CdTe 300K hybrid photon counting (HPC) detector from Dectris (Figure 2).

Results

The results in the table below illustrate the improved data quality and final refinement parameters that can be obtained with the new XtaLAB Synergy-S system, for highly absorbing crystal sample measurements.

Table 1: Experiment and refinement details for a highly absorbing sample with empirical formula: $[Pb_{32}O_{20.58}]\ (AsO_4)_2\ [(Si_{0.97}S_{0.10}As_{0.10}V_{0.38}Mo_{0.24})\ O_4]_2\ Cl_{9.77}$

	SuperNova with Atlas S2	XtaLAB Synergy-S with P300K CdTe
Total time (hours)	16	15
Exposure time (s)	240	25
Resolution (Å)	inf → 0.56	inf → 0.56
I/σ	9.29	24.56
Reflections collected	**27312**	**294255**
Completeness (%)	87.3	99.8
Redund.	1.6	15.4
R_{int}	0.026	0.052
R_1 (%)	**5.5 %**	**2.9 %**
Largest peak/hole (eÅ^{-3})	4.113 / -4.589	6.50 / -6.83

Through the improved brightness of the PhotonJet™ Ag source along with the improved quantum efficiency and fast readout of the detector the results were improved vs. the original dataset.

- Overall experiment time was reduced
- Shorter exposure times were possible
- More than 10 times the number of reflections were collected and thus higher redundancy was achieved
- Significantly higher I/σ was obtained
- R_1 was significantly improved

- For the new dataset the mixed metal oxide sites for Hereroite were able to be determined with greater certainty.
- Better bond precision indicated that the disordered oxygen atoms of the Si, Mo mixed site and their respective bond lengths confirms the presence of the two metals.
- Whilst still very good, the higher R_{int} value was a consequence of the fact that 10 times the number of reflections are being merged. Specifically in the higher resolution shells many more reflections were collected. Overall, the structural quality is vastly improved.

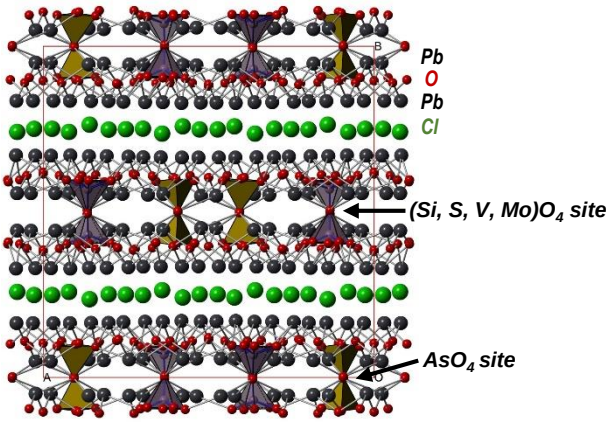

Figure 3. Structural model of hereroite

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